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Part II

THE ROLE OF THE GRAIN BOUNDARY IN THE DEFORMATION OF CERAMIC MATERIALS

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FOREWORD

This report was prepared by Materials Research Corporation, Orangeburg, New York under USAF Contract AF 33(616)-7961. This contract was initiated under Project No. 7350, "Refractory Inorganic Nonmetallic Materials", Task No. 735003, "Refractory Inorganic Nonmetallic Materials: Theory and Mechanical Phenomena". The work was administered under the direction of the Metals and Ceramics Laboratory, Directorate of Materials and Processes, Aeronautical Systems Division, with Mr. Gordon Atkins acting as project engineer.

This report covers work conducted from January 1962 to January 1963.

ABSTRACT

The mechanical behavior of MgO grain boundaries at elevated temperatures was investigated. The shear strength of bicrystal specimens was obtained (constant loading rate tests) at 1400°C for a wide range of crystal misorientations. Large twist, low tilt boundaries fractured at extremely low stress ~~(~150 g/mm²)~~ compared to other misorientations. For a given tilt angle the fracture stress decreased with increasing twist component; the decrease being sharper for low ~~(≤ 20°)~~ tilt boundaries and more gradual for boundaries possessing large tilt components. The effect of temperature on the grain boundary fracture stress was investigated for varying misorientations in the range 1200°-1500°C. For each type of boundary orientation there was a characteristic temperature where there was a sudden drop in strength, this transition temperature being lowest for boundaries with a high twist component. Experiments on tricrystals indicated that the weaker twist-type boundaries controlled the fracture strength of the entire body.

This technical documentary report has been reviewed and is approved.

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I. INTRODUCTION

It has been widely recognized (1)* that in metals at temperatures above approximately 0.5 of the melting point in degrees absolute, the grain boundaries play a predominant role in the creep and fracture process. In comparison to metals, very little consideration has been given to the role of the grain boundary in the process of elevated temperature fracture behavior of ceramic materials.

Studies on polycrystalline alumina (2,3,4) and magnesia (2,3) have indicated sharp changes in internal friction and elastic modulus with increasing temperature. These phenomena have been attributed to grain boundary effects. Other studies on high temperature creep of polycrystalline alumina (5,6) have indicated that grain boundary sliding may have occurred. All of these observations were of an indirect nature. However, direct observations of grain boundary sliding in bicrystals of sodium chloride and magnesia were obtained recently by Adams and Murray (7) at temperatures above 0.5 Tm (Tm = absolute melting point) which showed conclusively that elevated temperature grain boundary sliding does occur in the deformation of ceramic materials. In a subsequent study by Murray, et al, (8), it was shown that sliding did play a dominant role in the high temperature fracture process. It appeared that sliding and fracture along the bicrystal boundary were the same process, the only difference being the presence or absence of restraining jogs.

The present work was designed to extend the earlier findings (7,8), in particular to fully determine the effect of boundary misorientation on the intergranular fracture stress over a wide temperature range (>0.5 Tm) and to correlate these findings with the elevated temperature behavior of polycrystalline bodies.

II. EXPERIMENTAL PROCEDURES

A. Material

Random size lumps of MgO were obtained from the Norton Company. The impurity content of the material was quoted as 2000 ppm SiO_2, 1500 ppm Fe_2O_3, 2000 ppm CaO, 3500 ppm Al_2O_3,

*Numbers in parentheses indicate References.

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and 15 ppm B.

Bicrystal and tricrystal specimens were cut with abrasive wheels to the approximate shape and size desired from the large lumps which contained several grains. These specimens were then further abraded to final size, carefully polished in hot phosphoric acid, rinsed in hot water, and then rinsed in alcohol and thoroughly dried. The bicrystal specimens were approximately 3/16" X 1/8" X 5/16" in dimension with the grain boundary at a 45° angle to the longest specimen dimension, as shown in Figure 1.

B. Testing Procedure

The fracture strength of the specimens was determined in compression at various temperatures. The specimens were loaded between a fixed alumina pedestal attached to a rigid base plate and an alumina plunger, as shown in Figure 2. The load was transmitted to the plunger by a lever arm attached to a shaft operated by a 1/50 HP constant speed motor with appropriate reduction gears and measured with a dynamometer. The bicrystal grain boundary was oriented at a 45° angle to the stress direction so as to be subjected to a maximum shear stress. The stress reported, unless otherwise noted is the resolved shear stress along the boundary plane. The specimens were heated with a molybdenum wound tube furnace, the whole apparatus being enclosed by a water-cooled metal bell jar inside which an argon atmosphere was maintained.

C. Determination of Bicrystal Misorientation

Most of the bicrystal misorientations were determined by X-ray Läue patterns. However, for preliminary tests some were approximated by examination of the cleavage planes. The misorientation is reported in terms of twist and tilt components. The notation used for defining the misorientations in these terms was described in detail in the First Annual Report (January, 1962). This notation schematically shown in Figure 3, is as follows:

Two coordinate systems (x,y,z) and (x',y',z') are defined, each coinciding with the $\langle 100 \rangle$ direction respectively of the two crystals. The two origins fall on the bicrystal boundary and coincide. A perpendicular to the boundary is drawn through this common origin and the axis forming the smallest angle with the perpendicular to the boundary is defined as the z-axis. The $\langle 100 \rangle$ axis of the second crystal forming the smallest angle with this z-axis is defined as the z'-axis. The angle z-z' is defined as the angle of tilt. The primed coordinate system is then tilted through the angle z-z' to make the z-axes coincide. The x' and y' axes now fall in the x-y plane. The two angles x-x' and y-y' are equal and are defined as the angle of twist.

III. RESULTS

A. Fracture Stress - Crystal Misorientation Study

It became evident early in the course of this investigation that the stress for intergranular fracture (and/or sliding) was strongly misorientation dependent. In order to fully document this effect, the fracture stress as a function of misorientation was determined for numerous specimens of widely varying misorientations at a test temperature of 1400°C. At this temperature fracture was found to be intergranular in all cases. All specimens used were free of jogs, voids or any other grain boundary irregularities. The results of all experiments performed at this test temperature are listed in Table I and summarized in Figure 4. For convenience in presentation the results were grouped into two categories - those with tilt components greater than 30° and those with less than 20° tilt. It can be seen that the large twist, low tilt boundaries slid and fractured at extremely low stresses compared to other misorientations. Boundaries with low twist and either a low tilt or a high tilt were very strong and fractured at about 8,000 - 12,000 g/mm^2. This fracture stress decreased with increasing twist angle; the decrease being sharp for boundaries which had a low tilt (i.e., $\leq 20°$) and somewhat less sharp for boundaries with a large ($\gtrsim 30°$) tilt component. Attempts were made to find other features of the boundary misorientation that might elucidate the origin of this misorientation-fracture stress relationship. For example, as listed in Table I, the deviation of the boundary plane from the cube (cleavage) plane of each crystal was determined and examined. No particular trend was noted with respect to the fracture stress. Likewise, the fracture stress could not be correlated with any low indice crystallographic plane when such plane was near the boundary plane.

B. Fracture Stress - Temperature Study

The effect of temperature on the grain boundary fracture stress was investigated for varying misorientations in the range 1200°-1500°C. The results were obtained for three different and representative boundary misorientations and are shown in Figure 5. Curve A was obtained for a low twist (7°), high tilt (50°) boundary, curve B for a low twist (5°), low tilt (9°) boundary, and C for a high twist (35°), medium tilt (18°) boundary. For each specific misorientation bicrystal specimens were prepared from the same MgO block in order to minimize the variation in impurity content. In all cases, the fracture was intergranular and the stress computed is that in shear along the boundary. (A few tests were conducted at lower temperatures, e.g., 1100°C, and in almost all cases showed transcrystalline failure).

For bicrystal A, with a low twist, high tilt misorientation, the grain boundary fracture stress was found to change very slightly

(13,000 to 11,000 g/mm^2) from 1275° to 1400°C, after which there was a catastrophic drop in fracture stress. With bicrystal B, a low twist, low tilt boundary, a significant change in fracture stress occurred at a lower temperature. For bicrystal C, which possessed a 35° twist, 18° tilt boundary, the fracture stress dropped sharply at a lower temperature, namely 1200°C. These results indicate that the change in fracture stress with respect to temperature for grain boundaries in MgO bicrystals is strongly misorientation dependent, and that for each type of boundary misorientation there exists a characteristic temperature where a catastrophic drop in fracture stress takes place. This transition temperature is the lowest for boundaries with a high twist component.

C. Behavior of Grain Boundaries in Tricrystals

To correlate the findings of the role of the grain boundary in bicrystal specimens to the more complex case of polycrystalline bodies, the investigation was extended to tricrystal specimens.

A series of experiments were carried out on tricrystal systems in which each of the three grain boundaries had distinctly different misorientations. The objective here was to compare the fracture behavior of boundaries in a tricrystal specimen with an identically misorientated boundary in a bicrystal specimen.

Bicrystal specimens were cut from each of the three boundaries of a tricrystal block such that the boundaries were identically inclined to the direction of compression in both the bicrystal and tricrystal specimens (cut from the same block). This approach also eliminated all secondary effects, such as impurity content, grain boundary irregularities, etc. The results obtained are discussed below in detail.

The configuration of the grain boundaries in the first tricrystal system (block A) is shown in Figure 6. The results obtained on the bicrystal and tricrystals from this block are summarized in Table II. All fracture stress tests were carried out at 1350°C.

The experiments on bicrystal specimens indicated that of the three boundaries B/A was the weakest with a fracture stress of 4200 g/mm^2, A/C had an intermediate fracture stress of 5530 g/mm^2 and B/C was the strongest with a fracture stress of 12,080 g/mm^2. All three bicrystals slid uncontrollably to intercrystalline fracture.

In run #1 on this tricrystal the grain boundary B/A was found to be fractured at a shear stress as low as 2690 g/mm^2 as computed from the external load. This is presumably due to the fact that a slight amount of sliding occurred at some critical stress lower than the fracture stress as determined on the bicrystal test. The presence of the triple point then prevented further sliding and created a stress concentration which resulted in the rupture of the entire grain boundary. What is more significant is that the grain boundary B/C, on which the shear stress corresponding to external load was 1850 g/mm^2 and which had a bicrystal fracture strength of 12,080 g/mm^2, was also found to be fractured. Grain boundary A/C, which was stressed to 3850 g/mm^2 (shear stress as computed from external load) was not fractured and only had a small wedge shaped crack near the triple point. Further, a transgranular crack originated from the triple point into grain C along the (100) cleavage plane. This is shown in Figure 7.

A repeat run (#2, Table II) was similar to the first run and essentially verifies the above results that the strong grain boundary B/C can be fractured at low stresses as a result of fracture in the weaker boundary B/A. In this test the transgranular cleavage crack was not observed. To definitely establish whether this boundary B/C was being fractured because of some component of the original external load or due to the fracture of the neighboring weak boundary B/A, a third tricrystal was cut with B/C normal to the direction of compression but oriented in the same direction to the B/A as in other specimens (Figure 8). In this run (3a) when B/A was subjected to a shear stress of 2550 g/mm^2 a small crack was observed to have been initiated along the boundary with no fracture on B/C or A/C. However, on subsequent reloading (run 3b) B/A was stressed to 2860 g/mm^2 and a crack was initiated in boundary B/C. Boundary A/C which had a corresponding shear stress of 1320 g/mm^2 was intact. On the third loading which increased the stress on B/A to 3890 g/mm^2 (run 3c) and on A/C to 2580 g/mm^2, grain boundary B/C became completely fractured while A/C still remained intact and a transgranular crack originated from the triple point into grain C similar in direction to that in tricrystal #1. This is shown in Figure 9.

To further substantiate the above observations, a second boundary configuration, "B", was investigated at a test temperature of 1400°C. The configuration of the grain boundaries in the tricrystal (block B) is shown in Figure 10. The bicrystals were cut from each of the three boundaries of the tricrystal, as in the previous block. The results on the bicrystals and tricrystals are summarized in Table III.

The bicrystal experiments indicate that boundary C/B is the weakest with a fracture stress of 2990 g/mm^2, A/B has a fracture stress of 5050 g/mm^2 and C/A has a fracture stress of 9200 g/mm^2. All three bicrystals fractured intergranularly.

In tricyrstal test #1, boundary C/B was subjected to a shear stress equal to its fracture strength as obtained from bicrystal tests (i.e., 2990 g/mm^2). Not only was C/B found to be fractured, but the entire grain B was separated from the specimen indicating complete fracture along boundary A/B as well. This is shown in Figure 11. The shear stress as computed from the external load on A/B was 3640 g/mm^2 while its fracture strength from bicrystal tests was 5050 g/mm^2. Boundary C/A which had a corresponding shear stress (computed from external load) of 4240 g/mm^2 was not fractured.

In test #2 (Table III), boundary C/B was found to be fractured at a shear stress as low as 1500 g/mm^2 (nearly half its bicrystal fracture strength) as computed from the external load. This could be a result of some sliding that occurred at a critical stress lower than the fracture stress as determined on the bicrystal test. The boundary A/B was also found to be fractured, although the shear stress on it as computed from the external load was only 1538 g/mm^2. Presumably the presence of the triple point prevented further sliding of C/B creating a stress concentration which resulted in the rupture of boundary A/B. Grain boundary C/B which was stressed to 4240 g/mm^2 (shear stress as computed from external load) was not fractured. This is shown in Figure 12.

In test #3 (Table III) when the boundary C/B was subjected to a shear stress of 1191 g/mm^2 (computed from the external load) fracture was observed to have been initiated along the boundary. The grain boundary A/B which had a corresponding shear stress of 1210 g/mm^2 was found to be fractured. While C/A with a shear stress (computed from external load) of 1310 g/mm^2 was intact. This is shown in Figure 13.

These tests indicate that the smallest amount of sliding on the boundary C/B is sufficient to set up a stress concentration at the triple point which is high enough to rupture the boundary A/B. In all three tests the boundary C/A was nearly normal to the boundary C/B and may not have been subjected to any significant stress concentration.

IV. DISCUSSION

The elevated temperature shear strength of magnesia grain boundaries has been shown to be strongly orientation dependent. The notation employed here to describe the boundary misorientation does not define the direction of the boundary through the bicrystal matrix but merely utilizes the boundary plane as a reference plane from which to describe the relative orientations of the two crystal lattices. Thus the twist and tilt connotation only refers to an atomistic twist and tilt configuration when the boundary approaches either a pure twist or pure tilt type boundary. Nevertheless, the data show that high twist, low tilt boundaries (as defined here) are much weaker than boundaries of other misorientations and thus strongly suggest a dependency of the strength on the atomistic configuration of the boundary. It was not possible to obtain specimens of pure tilt and pure twist in order to experimentally check this implication. Specimen No. 3 (Table I) approaches a pure twist boundary and indeed possessed a low (500 g/mm^2) fracture strength. On the other hand, high twist specimens, e.g., No. 1,2,4 and 5, which possessed moderate tilt components, also exhibited low fracture stresses.

Although the boundary atomistic configuration, per se, is implied here as the characteristic most responsible for the low fracture stresses of certain misorientations, other factors cannot be overlooked. The tendency for grain boundary segregation of a particular solute could also be strongly misorientation dependent (a second phase was never detected at the boundary) and thereby result in a weak boundary. The inability of certain boundaries to transmit plastic deformation from one crystal to the other could also lead to premature failure.

The elevated temperature strength of magnesia grain boundaries has also been shown to be strongly temperature dependent as expected. However, this temperature dependency is manifested by a sharp discontinuity in the form of a sudden loss in strength at a temperature slightly above the temperature at which intercrystalline failure occurs (at lower temperatures transcrystalline failure is the rule). When one examines both the temperature and misorientation dependency it is found that the weaker twist type boundaries exhibit a lower temperature for the sudden fracture strength decrease.

The tricrystal experiments clearly show that the weaker twist type boundaries control the fracture strength of the entire body. In the case of a randomly oriented polycrystalline matrix the elevated temperature fracture strength of the body in all probability would be less than the fracture strength of the weakest boundary due to the onset of grain boundary sliding at some lower stress. It has been previously demonstrated (8) that in most cases sliding precedes fracture. The low fracture strength of some boundaries in the present tricrystal experiments can be explained in this manner. It thus appears that in practice if the temperature of application exceeds the "transition" temperature of the weakest boundary, fracture will occur at a very small stress, and due to the notch sensitivity of the material, propagate to cause catastrophic failure of the body. A sharp decrease in the strength of polycrystalline MgO around 1200°C that was accompanied by intercrystalline failure has been reported (9).

V. WORK IN PROGRESS

The work described here has been seriously hampered by the lack of a suitable supply of MgO material. In the random lumps obtained it has not been possible to find all the desired boundary misorientations. Furthermore, this material varies considerably in purity from lot to lot (and perhaps even from specimen to specimen). To further complicate matters the suppliers have recently ceased to make available quantities of these random size lumps at other than prohibitive costs.

In order to circumvent these problems, work is now in progress on high purity MRC prepared KCl bicrystals from controlled misorientation seeds using a modified Czochralski technique. This process consists of gripping a seed crystal in a water-cooled chuck and immersing its end in the liquid which is maintained slightly above its melting point. The crystal is then grown by slowly withdrawing the seed from the melt. By altering the chuck such that two seed crystals are held in a given orientation with respect to each other, a bicrystal of predetermined misorientation will result. A schematic of the apparatus is shown in Figure 14. The advantages are:

(a) By adjustment of the withdrawal rate of the seed crystals, bicrystals of almost any dimension can be produced.

(b) The bicrystal is not subjected to any constraining device which could lead to fracture or deformation.

(c) The substructure formation is considerably less than in the Bridgman method because of the absence of a mold.

(d) The procedure can be interrupted at any time without exposing the bicrystal to the danger of fracture.

(e) By equipping the water cooled chuck with a vernier protractor, any relative orientation can be obtained easily and accurately.

(f) The method is a purification step similar to the Bridgman method and results in crystals appreciably purer over most of their volume than the starting material.

To date several bicrystals of pure twist, pure tilt, and medium twist, medium tilt orientation have been grown. Tests to determine the fracture strength as a function of temperature and misorientation are in progress.

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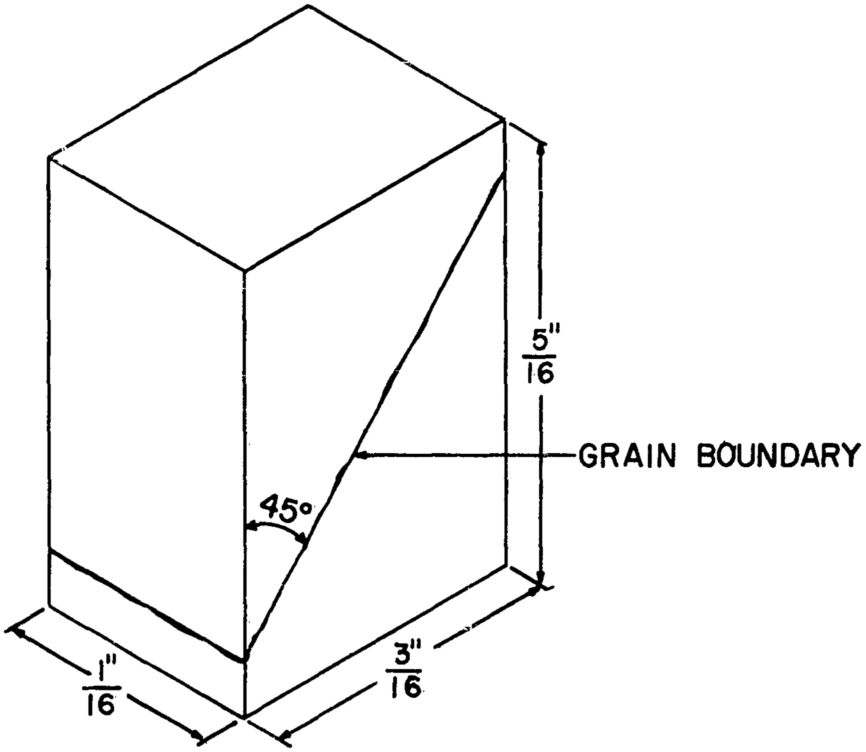

FIGURE I. FORM OF MgO BICRYSTALS USED IN THE TESTS.

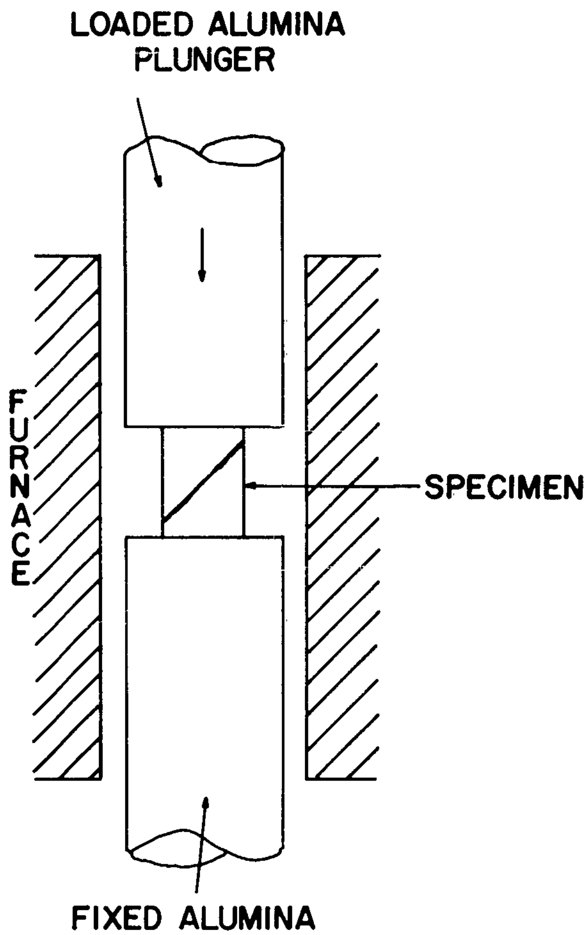

FIGURE 2. CREEP-RUPTURE TEST APPARATUS

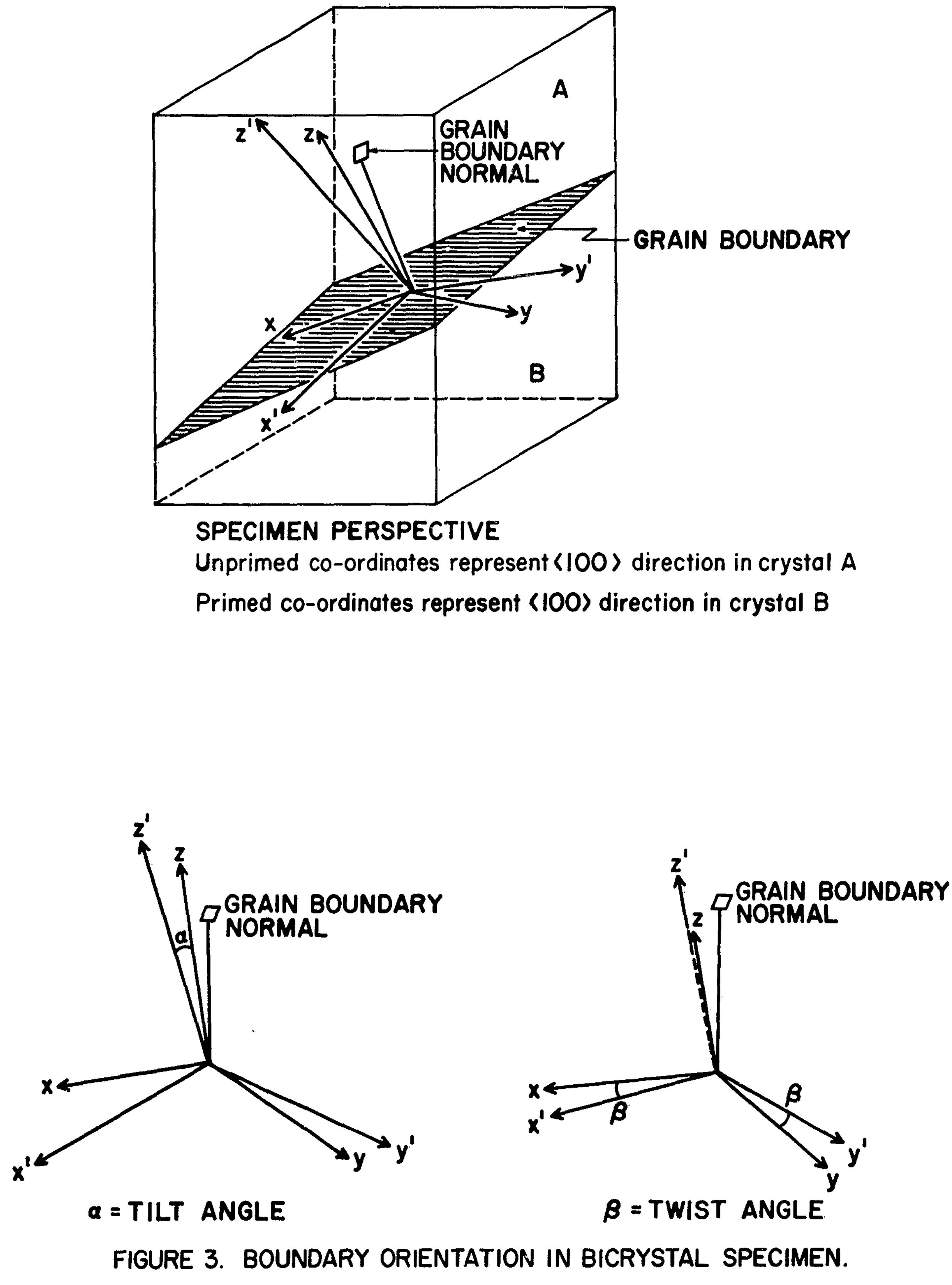

FIGURE 3. BOUNDARY ORIENTATION IN BICRYSTAL SPECIMEN.

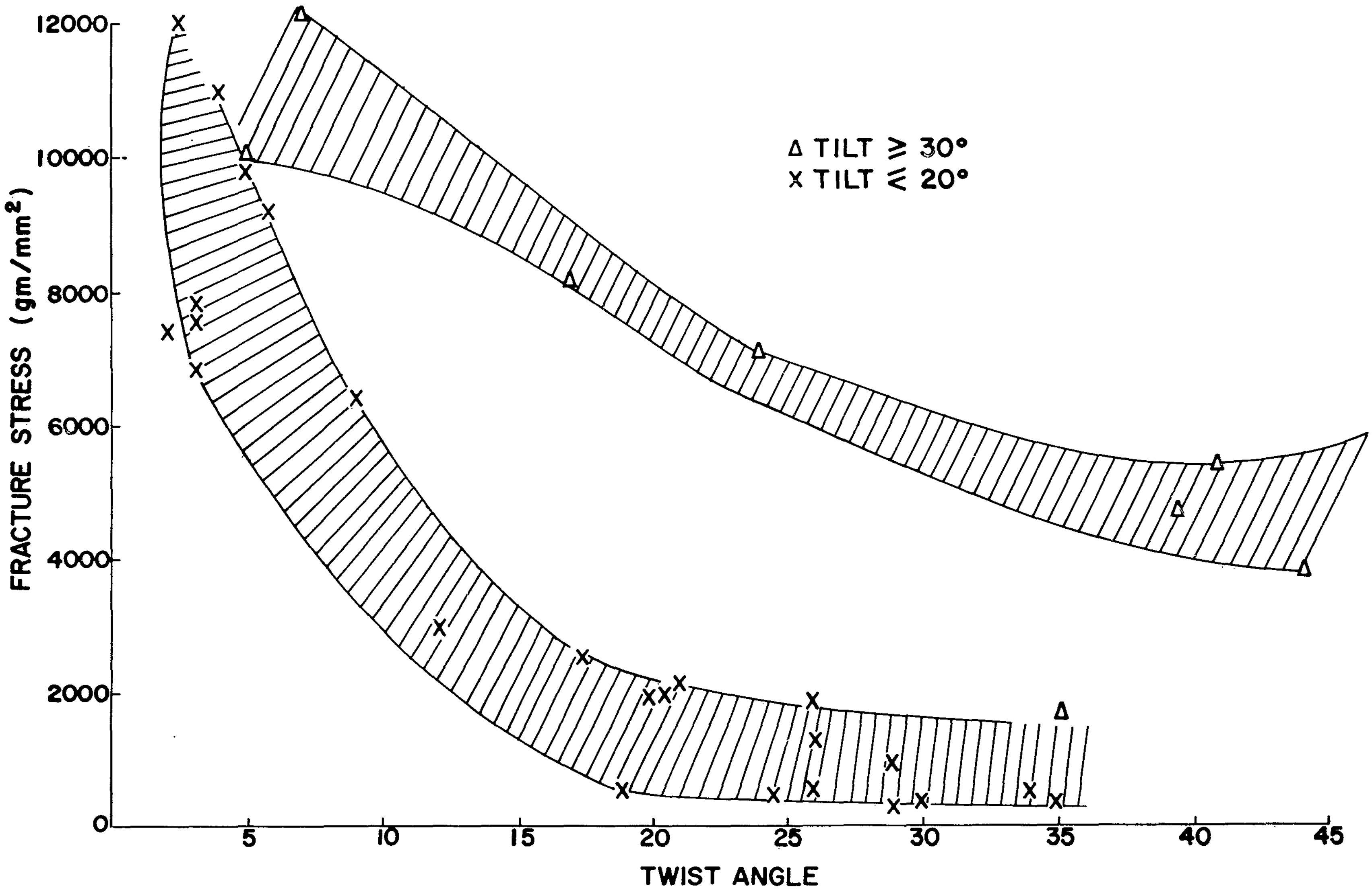

FIGURE 4. MgO BICRYSTAL GRAIN BOUNDARY FRACTURE STRESS IN SHEAR VS. TWIST ANGLE AT 1400° C.

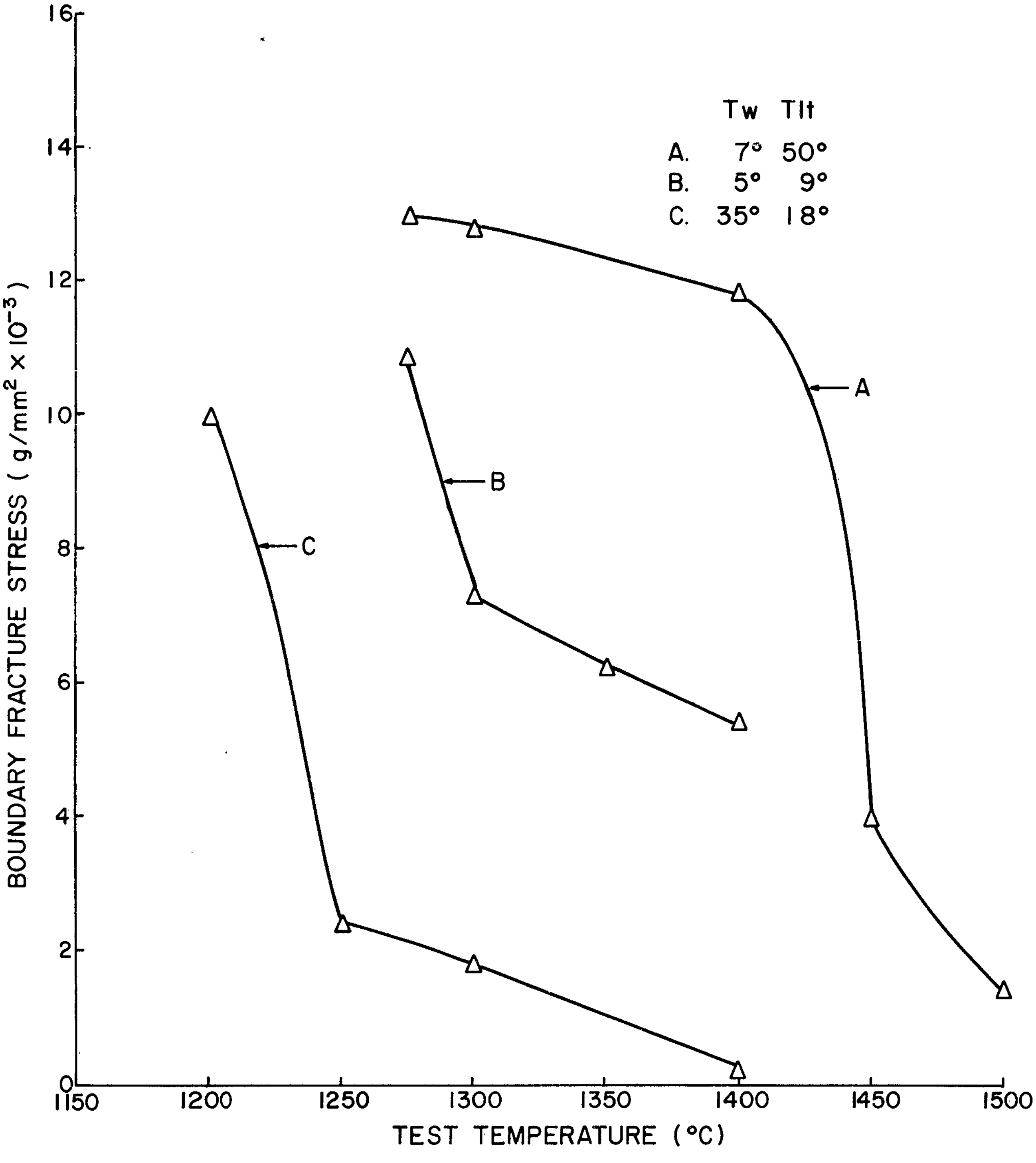

FIGURE 5. FRACTURE STRESS AS A FUNCTION OF TEMPERATURE FOR MgO BICRYSTALS

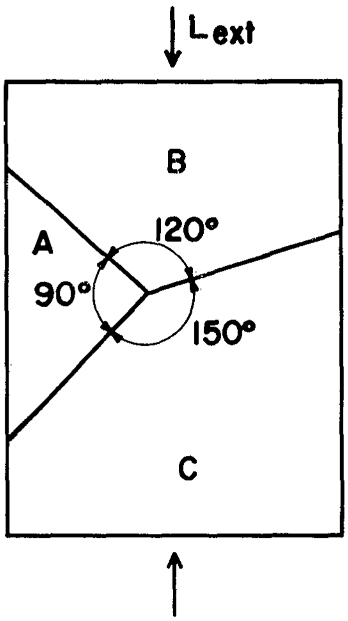

FIGURE 6. BOUNDARY CONFIGURATION IN TRICRYSTALS. (BLOCK A)

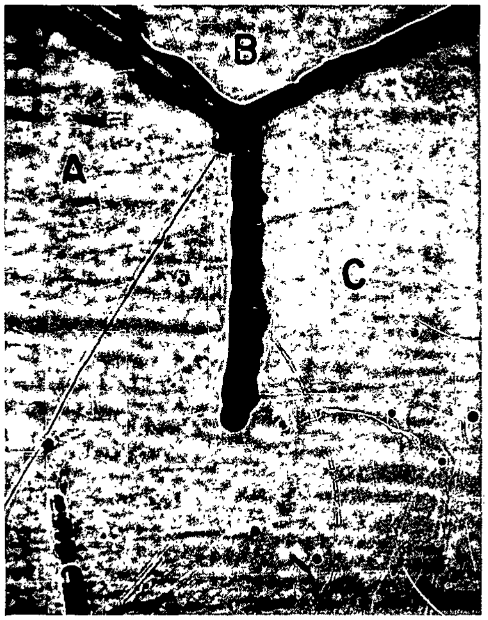

FIGURE 7. TRICRYSTAL AFTER TEST # I SHOWING BOUNDARIES B/A AND B/C FRACTURED WITH CLEAVAGE CRACK IN GRAIN C. (BLOCK A) MAG. X 250.

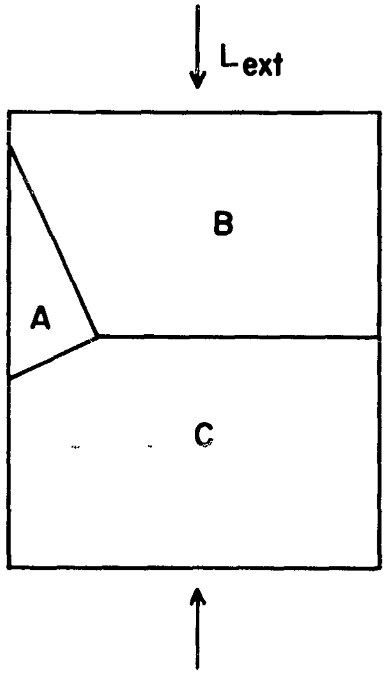

FIGURE 8. ORIENTATION OF GRAIN BOUNDARIES TO DIRECTION OF COMPRESSION AXIS IN TRICRYSTAL TEST #3. (BLOCK A)

FIGURE 9. TRICRYSTAL AFTER TEST # 3(C) SHOWING BOUNDARIES B/A AND B/C FRACTURED WITH CLEAVAGE CRACK IN GRAIN C. (BLOCK A) MAG. X250.

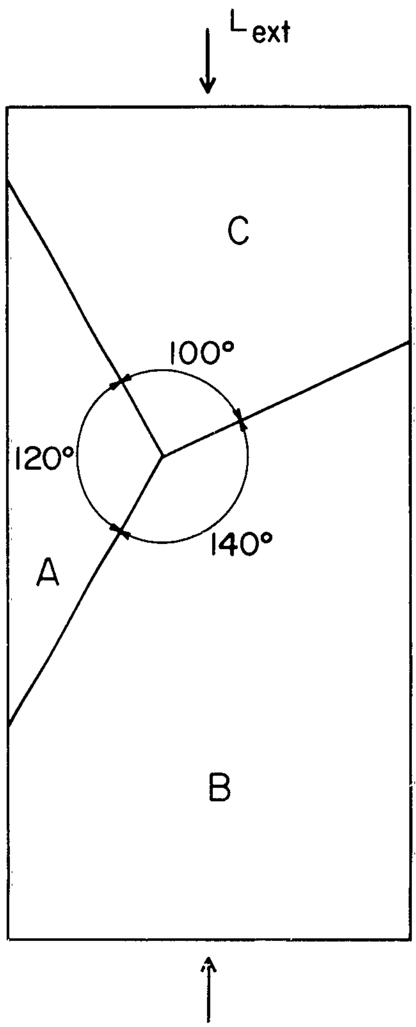

FIGURE 10. BOUNDARY CONFIGURATION IN TRICRYSTALS (BLOCK B)

Figure 11 - Tricrystal test #1, showing boundaries C/B and A/B fractured with complete separation of grain B (block B).Mag. X10

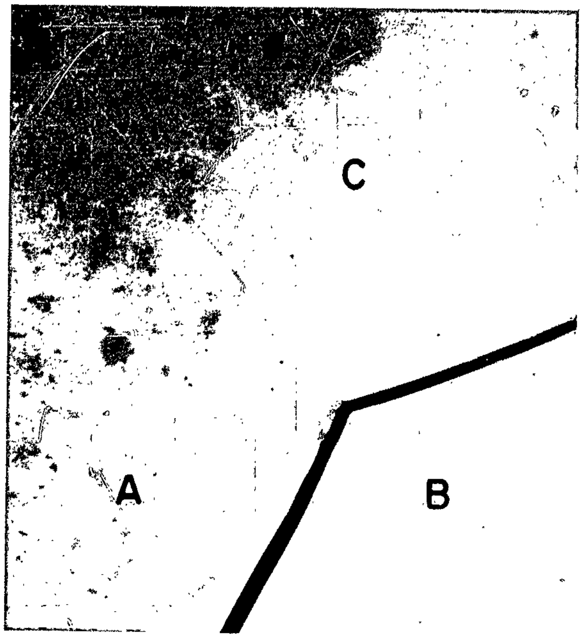

Figure 12 - Tricrystal test #2, showing boundaries C/B and A/B fractured (block B). Mag. X250

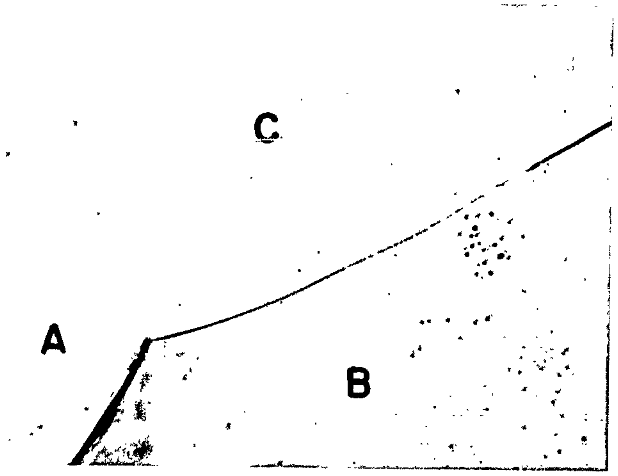

Figure 13 - Tricrystal test #3, showing boundary C/B fractured in parts and A/B fractured completely (block B). Mag. 250X

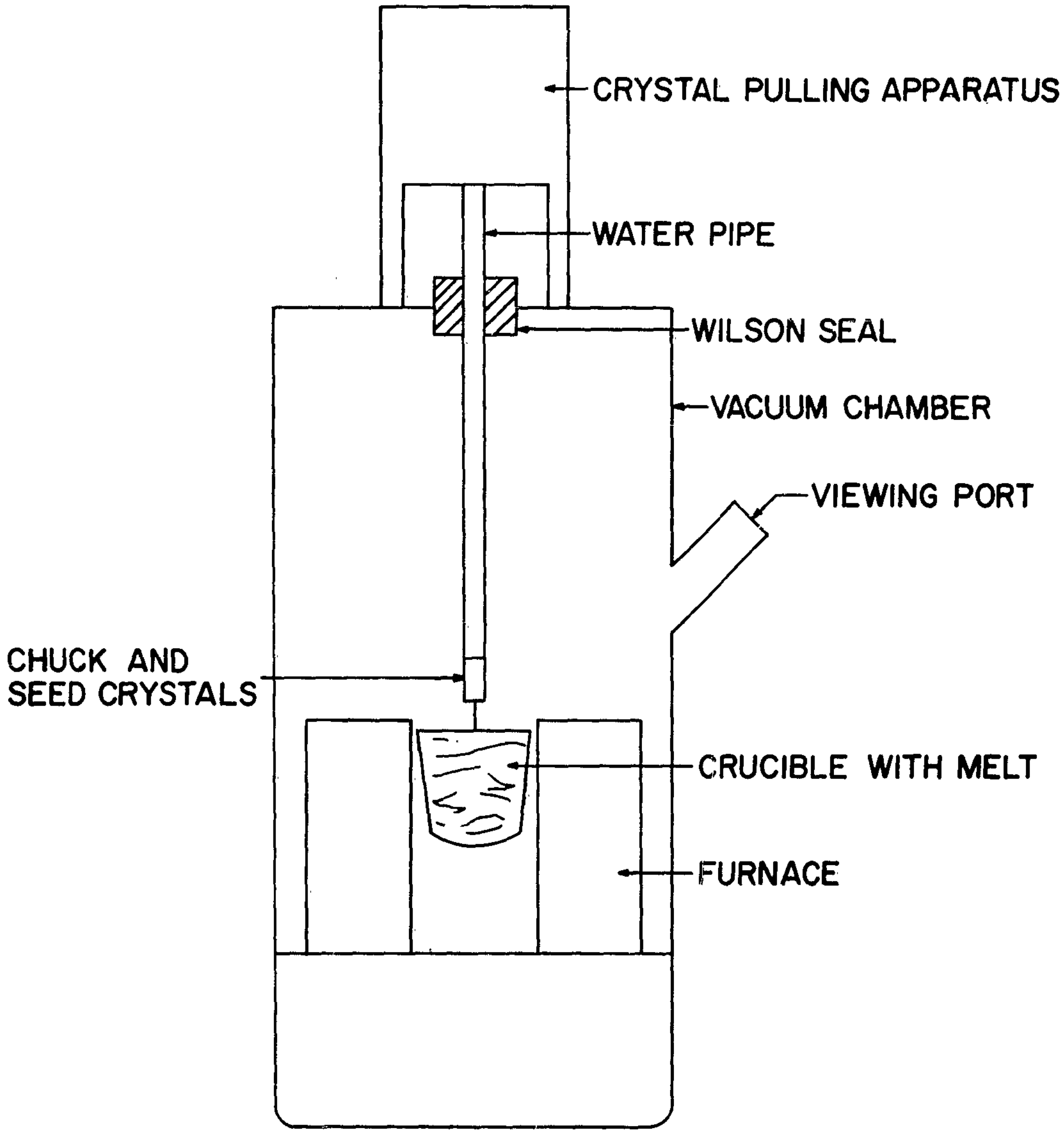

FIGURE 14. CRYSTAL PULLING APPARATUS

TABLE I

EFFECT OF MISORIENTATION ON THE GRAIN BOUNDARY FRACTURE STRESS OF MgO BICRYSTALS AT 1400°C

Spec. No.	Twist	Tilt	θ_A*	θ_B*	Fracture Stress (g/mm^2)
1	24°	21°	12°	15°	220
2	30	12	6	12	190
3	19	6	9	10	500
4	26	15	36	43	600
5	29	17	14	18	700
6	44	41	26	29	3,800
7	21	10	7	16	2,150
8	5	39	29	32	10,000
9	4	3	1	2	10,950
10	3	13	25	32	7,610
11	7	50	16	43	12,150
12	26	22	28	22	3,240
13	35	18	12	23	300
14	34	20	17	22	497
15	5	6	35	38	9,840
16	17	12	25	38	2,495
17	39	42	9	38	5,220
18	2	3	13	14	12,000
19	41	44	13	39	6,000
20	26	14	38	42	2,600
21	3	15	2	13	6,730
22	24	43	41	44	7,320
23	3	8	40	41	7,825
24	2	15	42	33	7,360
25	20	12	14	17	1,775
26	17	35	25	30	8,250
27	9	8	13	18	6,440
28	6	10	16	26	9,200
29	20	12	14	17	1,975
30	26	15	36	43	600

* θ represents the smallest angle between the grain boundary pole and the [100] pole in grains A and B of the bicrystal.

TABLE II

STRENGTH OF GRAIN BOUNDARIES IN BICRYSTAL
AND
TRICRYSTAL SPECIMENS AT 1350°C FROM BLOCK "A"

Grain Boundary	B/A Tw. 20° Tlt. 12°	B/C Tw. 30° Tlt. 51°	A/C Tw. 23° Tlt. 52°
Fracture Stress (from bicrystal test)	4220 g/mm^2	12,080 g/mm^2	5530 g/mm^2
Tricrystal Run #1 Shear Stress on Boundary	2690 g/mm^2 g.b. fractured	1850 g/mm^2 g.b. fractured	3850 g/mm^2 g.b. intact except for small wedge shaped crack near triple pt. - trans-granular crack from triple pt. into C
Tricrystal Run #2 Shear Stress on Boundary	1910 g/mm^2 g.b. fractured	1645 g/mm^2 g.b. had cracks and voids	2845 g/mm^2 small wedge shaped crack near triple pt. (as in #1) no trans-granular crack
Tricrystal Run #3 (a) Shear Stress on G.B.	2550 g/mm^2 crack initiated at edge of g.b.	no shear stress slight crack near g.b. edge	530 g/mm^2 g.b. intact
(b) Shear Stress on G.B.	2860 g/mm^2 g.b. fractured	no shear stress g.b. fractured continuously near triple pt. and intermittently at edge	1320 g/mm^2 g.b. intact
(c) Shear Stress on G.B.	3890 g/mm^2 g.b. fractured	no shear stress g.b. fractured	2580 g/mm^2 g.b. intact

TABLE III

STRENGTH OF GRAIN BOUNDARIES IN BICRYSTAL
AND
TRICRYSTAL SPECIMENS AT 1400°C FROM BLOCK "B"

Grain Boundary	A/B Tw. 3° Tlt. 22°	C/B Tw. 14° Tlt. 3°	C/A Tw. 6° Tlt. 10°
G.B. Fracture Stress (from bicrystal test)	5050 g/mm^2	2990 g/mm^2	9200 g/mm^2
Tricrystal Test #1 Shear Stress on Boundary	3640 g/mm^2 g.b. fractured	2988 g/mm^2 g.b. fractured, grain boundary completely separated from spec.	4240 g/mm^2 g.b. intact
Tricrystal Test #2 Shear Stress on Boundary	1538 g/mm^2 g.b. fractured	1500 g/mm^2 g.b. fractured	1488 g/mm^2 g.b. intact
Tricrystal Test #3 Shear Stress on Boundary	1210 g/mm^2 g.b. fractured	1191 g/mm^2 g.b. fractured in parts	1310 g/mm^2 g.b. intact

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Unclassified Report

The mechanical behavior of MgO grain boundaries at elevated temperatures was investigated. The shear strength of bicrystal specimens was obtained (constant loading rate tests) at 1400°C for a wide range of crystal misorientations. Large twist, low tilt boundaries fractured extremely low stress ($\sim$ 150 g/mm^2) compared to other misorientations. For a given tilt angle the fracture stress decreased with increasing twist component; the decrease being sharper for low (< 20°) tilt

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boundaries and more gradual for boundaries possessing large tilt components. The effect of temperature on the grain boundary fracture stress was investigated for varying misorientations in the range 1200° - 1500°C. For each type of boundary orientation there was a characteristic temperature where there was a sudden drop in strength, this transition temperature being lowest for boundaries with a high twist component. Experiments on tricrystals indicated that the weaker twist-type boundaries controlled the fracture strength of the entire body.

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